PROCESSING REQUIREMENTS FOR SLIP-CAST FUSED SILICA RADOMES

Technical Report No. 2

June 1972

By

J. N. Harris
E. A. Welsh

Prepared Under Contract N00017-70-C-4438

For

Naval Ordnance Systems Command
Weapons Dynamics Division (Code ORD-035)
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By

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ABSTRACT

This report consists of a process specification for slip-cast fused silica radomes. The rationale behind the requirements for this specification is given.

TABLE OF CONTENTS

This report contains 25 pages.

LIST OF ILLUSTRATIONS

I. PURPOSE

The purpose of Contract No. N00017-70-C-4438 is to perform research and development directed towards the development of techniques to fully exploit the potential of readily available ceramic systems for use as structural components in hypersonic missile applications.

Parts II and III of this report provide the rationale leading to the "Processing Specification for Slip-Cast Fused Silica Hardware" of Appendix I.

II. INTRODUCTION

The definition of a processing specification is more difficult than that for the raw materials. Sintering time-temperature is dependent on the starting material (i.e., synthetic fused silica or fused quartz). Different results are usually obtained in sintering slip-cast fused silica in gas fired and electrically heated kilns even though the same time-temperature firing schedule is followed. It is generally assumed that normally there is more moisture in the kiln atmosphere of a gas fired kiln than there is in an electrically fired kiln. If this is the case this could account for the difference in sintering rate. Less significant variations have even been seen between firings in different electrically heated kilns for the same time-temperature sintering conditions. Differences in moisture in the firing atmosphere, placement of the hardware to be fired in relation to the heating elements and placement of controlling thermocouples, within the kiln can be causes for variations in firing conditions between two kilns. Therefore, each manufacturer must establish a particular time-temperature sintering condition to produce the desired density and mechanical properties ahead of time. Guidelines are given in this report based on several years of experience at the Georgia Institute of Technology using several different kilns.

A processing specification can only place broad requirements on such items as mold materials, storage and handling of the fused silica slip, handling and proper drying of the freshly cast ware, and sintering to the final desired properties. These requirements, and the rationale behind their inclusion in a fused silica hardware processing specification are discussed in the following section.

III. PROCESS QUALIFICATION REQUIREMENTS

A. Mold Material

Fleming 1/ has reported that different plasters produce varying degrees of surface devitrification in slip-cast fused silica. However, he notes that these differences in surface devitrification seemed to have no effect on bulk devitrification or sintering behavior. For this reason, any commercially available pottery plaster should be suitable for use.

Inert materials such as porous ceramics and resin bonded grain have shown promise in high volume production areas such as sanitary ware and dinnerware production 2,3/. They are suitable for use in radome production, but introduce additional design problems, such as mold shrinkage during cure or sintering. Their use can only be recommended in cases where the longer mold life is sufficient to offset the greatly increased mold manufacturing cost.

B. Mold Release Agents

A mold release agent is a thin film between the mold and the fused silica casting. This film aids in removing the cast piece from the porous mold. Three mold release agents have been used at Georgia Tech, namely graphite, ammonium alginate, and sodium alginate. No reductions in strength have been observed with either but it is felt that the sodium alginate should be avoided since alkalies increase devitrification rates greatly 1/. As a general rule mold release agents should be avoided since they tend to produce uneven, rough surfaces. When used they should be held to as small a thickness as possible.

C. Slip Storage and Handling

Fused silica casting slip should be stored in polyethylene or similar polymeric containers, or in polyethylene lined metal drums. The slip should not be in contact with any metal. Containers should be continuously rolled or otherwise agitated to prevent settling. Slip should not be allowed to freeze since this apparently precipitates silicic acid from the slip, and prevents casting of a dense body. The slip generally contains a small portion of -4 +10 mesh material which is slightly smaller than the interstices in the grinding media bed. This material should be screened, usually to -40 mesh, at the time the plaster mold is filled. Slips should be used as soon as possible after milling. No aging problems have been noted with commercially available technical grade and high purity casting slips, but one manufacturer producing slip for in-house use has reported that the slip tends to gel after 3 to 4 weeks.

D. Slip Casting

Molds for slip-casting are usually prepared from pottery plaster using procedures recommended by the manufacturer. A plaster/water mix of 50/40 has been found best for slip-casting silica. For pressure casting, the walls of the plaster mold should be thick enough to withstand the internal pressure. For casting large shapes (up to 50-inch long radomes), a 3-inch plaster wall thickness has been satisfactory for pressures up to 20 psi. The outer contour of the mold should conform as closely as possible to the inner or casting surface. For example, an ogive radome could be cast in a conical mold or even a mold with its outer surface formed by portions of two cones.

The uniform wall thickness aids in obtaining a uniform wall thickness in the casting. In slip-casting, an aqueous suspension of finely ground particles (casting slip) is poured into a porous mold which defines the shape of the object to be produced. Capillary action draws water from the slip into the porous mold, increasing the solids content of the slip adjacent to the plaster, forming a solid wall. The water eventually evaporates from the mold. The thickness of the wall increases with the time the slip is left in the mold. When sufficient thickness has built up, the excess slip is drained out by vacuum or gravity leaving a dense but somewhat leathery shape in the mold.

The rate of wall build-up follows the parabolic filtration equation The expression

$$W = 0.048 \sqrt{\theta - 2.7} - 0.0207 \qquad (1)$$

where W = wall thickness in inches

θ = casting time in minutes

has proved satisfactory for estimating casting times at atmospheric pressure. Long casting times should be avoided due to problems such as settling of the slip in the mold. As an alternative, the slip may be pressurized and casting times drastically reduced. In this case the expression

$$W = 0.012 \sqrt{\theta P} - 0.047 \qquad (2)$$

where P = gage pressure - psi

may be used to estimate casting times. Figure 1 shows data for both expressions graphically. In either case, casting at atmospheric or at

increased pressure, the time for a given wall thickness may be estimated from a trial casting by the relationship

$$\frac{W_1^2}{W_2^2} = \frac{\theta_1}{\theta_2} \qquad (3)$$

where W_1 = wall thickness at time θ_1

W_2 = wall thickness at time θ_2.

In this way casting times necessary to produce a given wall thickness may be readily determined. These curves are generally accurate to within ± 10 per cent for slips with 7 micrometer (μm) mean particle diameters. Coarser slips will cast more rapidly while finer grinds will cast more slowly.

Since the coarse fractions of most fused silica slips settle to some extent, it is advisable to use pressure casting to hold casting times to a reasonable value, (i.e., 6 hours). From Equation (2), it can be seen that a wall of 0.97 inches roughly C-band can be reached in 6 hours at 20 psi pressure. X-band and higher frequency radomes could be cast in correspondingly shorter times.

E. Drying

Drying is a critical portion of the fabrication process, particularly for large shapes. At the end of casting, the pore spaces in the cast part are filled with water, and a thin film of water surrounds each particle. Since the particles are not in direct contact, but are cushioned by a water layer, the cast part has very little strength and will slump slightly if removed from the mold. At this point castings from slips with 7-8 μm mean particle

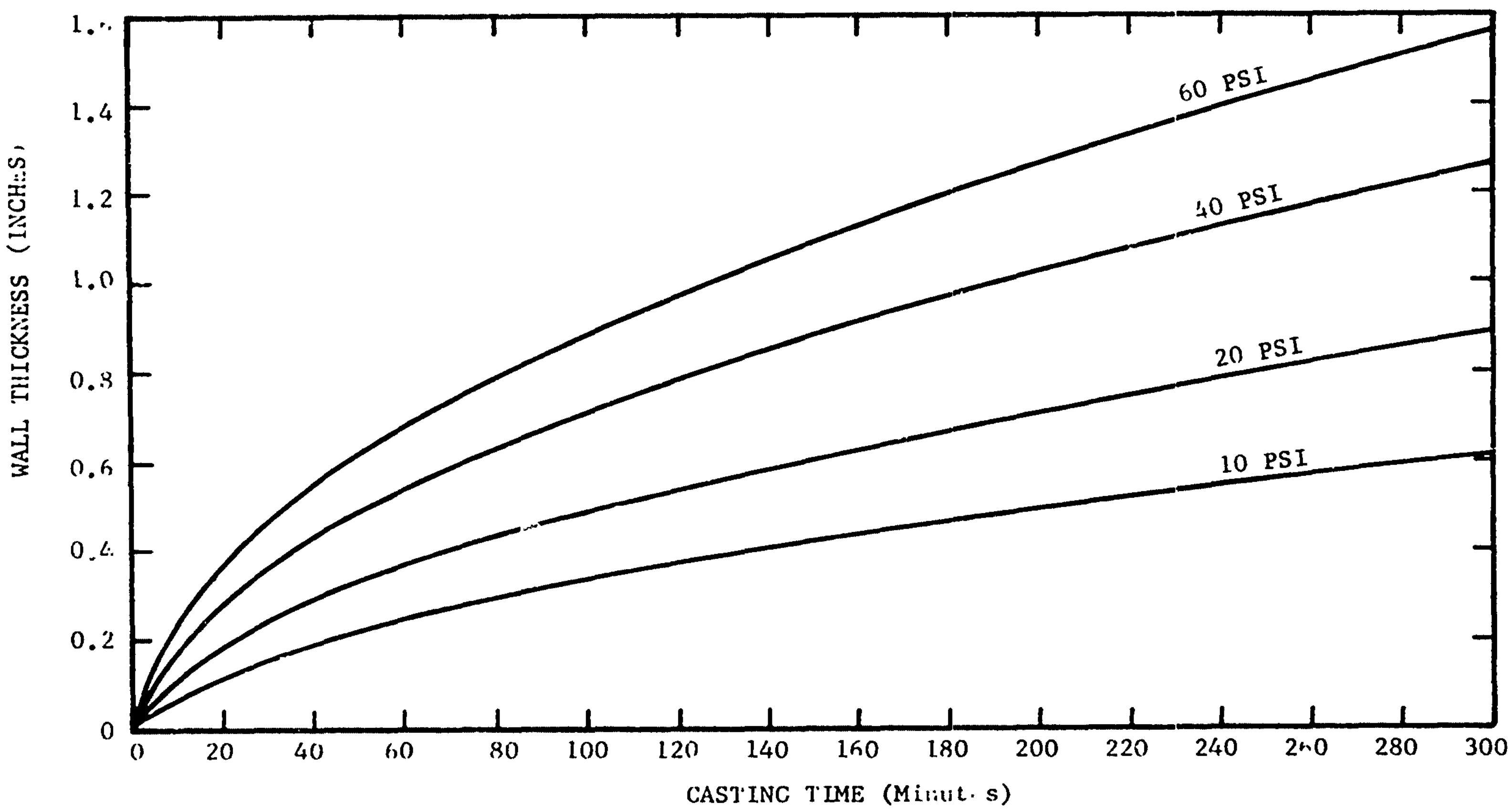

Figure 1. Cast Wall Thickness versus Time of Casting for Fused Silica.

diameter silica slips indicate drying shrinkages in the range from 0.05 to 0.1 per cent. Since these measurements were referenced to model size, rather than actual mold size, the linear drying shrinkage is more probably in the range from 0.2 to 0.4 per cent, assuming that the plaster mold is approximately 0.2 per cent oversize as a result of expansion on settling. This small shrinkage corresponds to a small moisture reduction on the order of 0.5 to 0.7 per cent. At this point, and certainly by the time 1.0 per cent moisture has been removed from the cast part, the part may be safely removed from the mold. It should be noted that since there is no convenient method of measuring the moisture content of the part in the mold, the simplest method of determining when the part may be safely removed from the mold is to measure the inside or outside diameter of the part from time to time. When shrinkage ceases, the part may be removed safely.

The two most important points in drying large slip-cast fused silica shapes are then,

(1) Drying should be carried out in the mold until the moisture content drops below the critical point (from 0.5 to 1 per cent loss).

(2) Drying rate during this time should be slow enough that moisture gradients across the cast wall are not large enough to cause cracking from differential shrinkage. These conditions are difficult to estimate or define from existing data and must be determined by trial and error for the particular part and mold configuration used. (Drying rate is also restricted by mean particle diameter. More finely ground slips will form cast parts with smaller and therefore less permeable pores.)

After the critical moisture content is reached, the part may be removed from the mold and dried more rapidly. Small shapes have been successfully dried at 350° F, but for most shapes it is recommended that drying temperatures be kept below 125° F. After the moisture content has been reduced to 1 or 2 per cent, the part should be dried to at least 250° F, and preferably 350° F for a minimum of 4 hours before sintering.

F. Sintering

Sintering is the most important single aspect of processing slip-cast fused silica hardware. The temperature and duration of sintering, along with the purity and particle size of the slip determine the physical properties of the finished item. During sintering particles tend to consolidate resulting in an increase in strength and bulk density. With increasing time the sintering rate slows until an end point density, directly proportional to sintering temperature is reached. At the same time, the vitreous silica is devitrifying to form cristobalite, which due to a phase change, causes a loss of strength when cooled to room temperature. At some point in sintering, the strengthening effect of sintering is balanced by the weakening effect of cristobalite growth and property values begin to fall.

Due to differences in particle size in slips, and sintering characteristics in furnaces, and properties required for finished hardware items, it is difficult to specify an optimum time and temperature for sintering. Therefore, sintering times and temperatures must be established by trial and error. Since this specification is concerned with hardware which is intended primarily for EM window or radome applications, the dielectric constant of

the sintered product must be maintained within specified limits. For a given purity of fused silica the dielectric constant is dependent entirely on density. Therefore, density seems to be a reasonable property choice for establishing sintering schedules. Figure 2 shows bulk density as a function of time and temperature for a typical fused silica slip of the type specified for radome production.

Test firings should be made on either full size shapes or similar shapes. For instance a radome may be approximated by a closed end cylinder. Although the maximum bulk density increases with temperature, within limits, properties such as Young's modulus and modulus of rupture, and dielectric constant are density dependent, as shown in Figures 3, 4, and 5, while others such as thermal expansion and specific heat are independent of density.

G. Thermal Gradients During Sintering

Boland 7/ has established guidelines for establishment of heating rates for slip-cast fused silica hardware. In this he establishes 20° F as the maximum temperature gradient tolerable in a cast part. From his sample problems, one can easily calculate both the soak time at 2000° F necessary to bring the backside temperature of a radome from room temperature to 1900° F, and the maximum permissible heating rates from 2000° F to the vicinity of 2200° F, which will allow no more than a 20° F lag in backside temperature. Plots of these functions with respect to wall thickness are shown in Figures 6 and 7. From these it is seen that the soak time at 2000° F is short enough to be ignored for wall thickness under 0.25-inch. Similarly, on heating to final temperature, the heating time becomes low enough for thick walled radomes, that a compromise maximum rate of 700° F

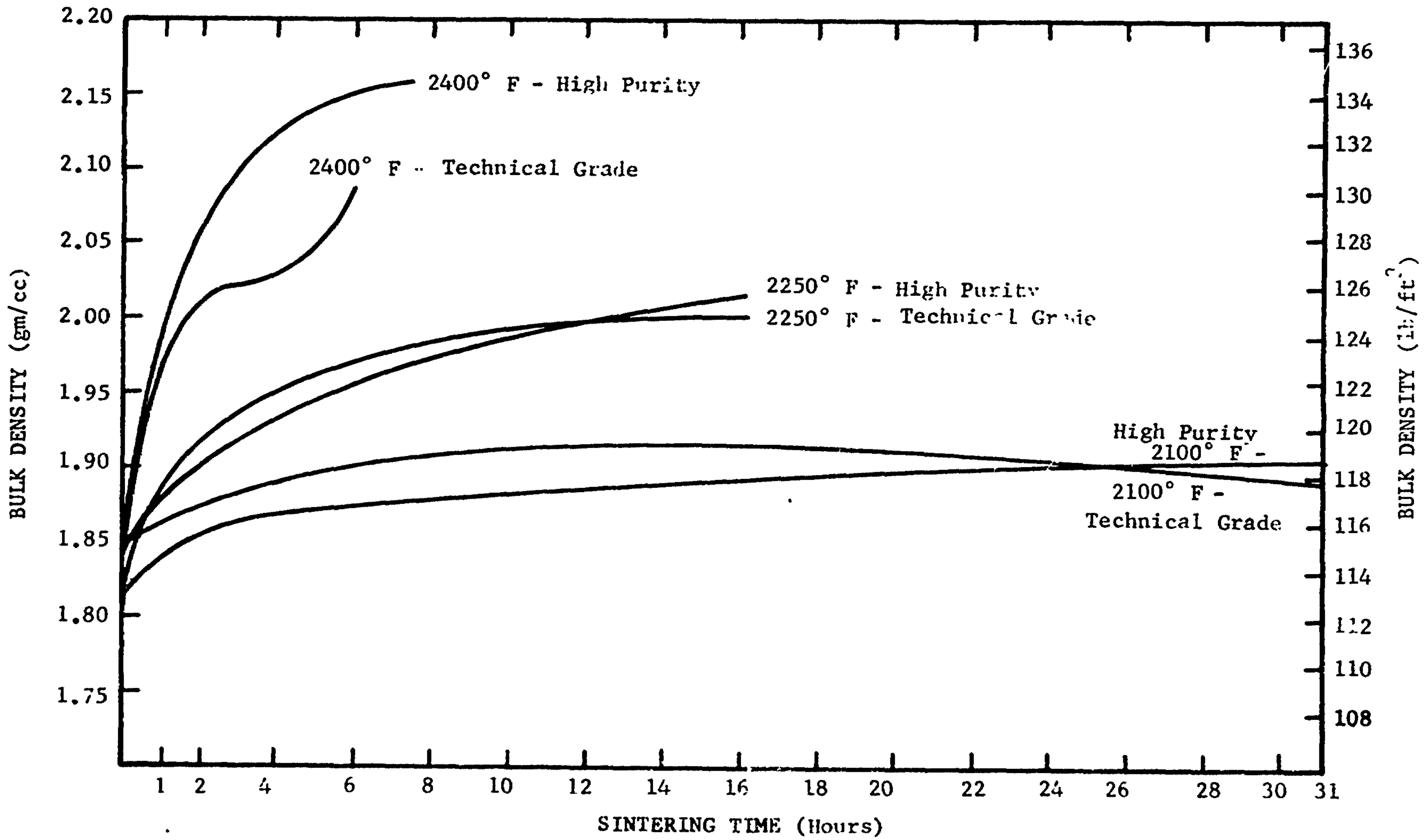

Figure 2. Bulk Density versus Sintering Time for High-Purity and Technical Grade Fused Silica Slips Sintered at 2100° F, 2250° F, and 2400° F. (Reference 4, page 18.)

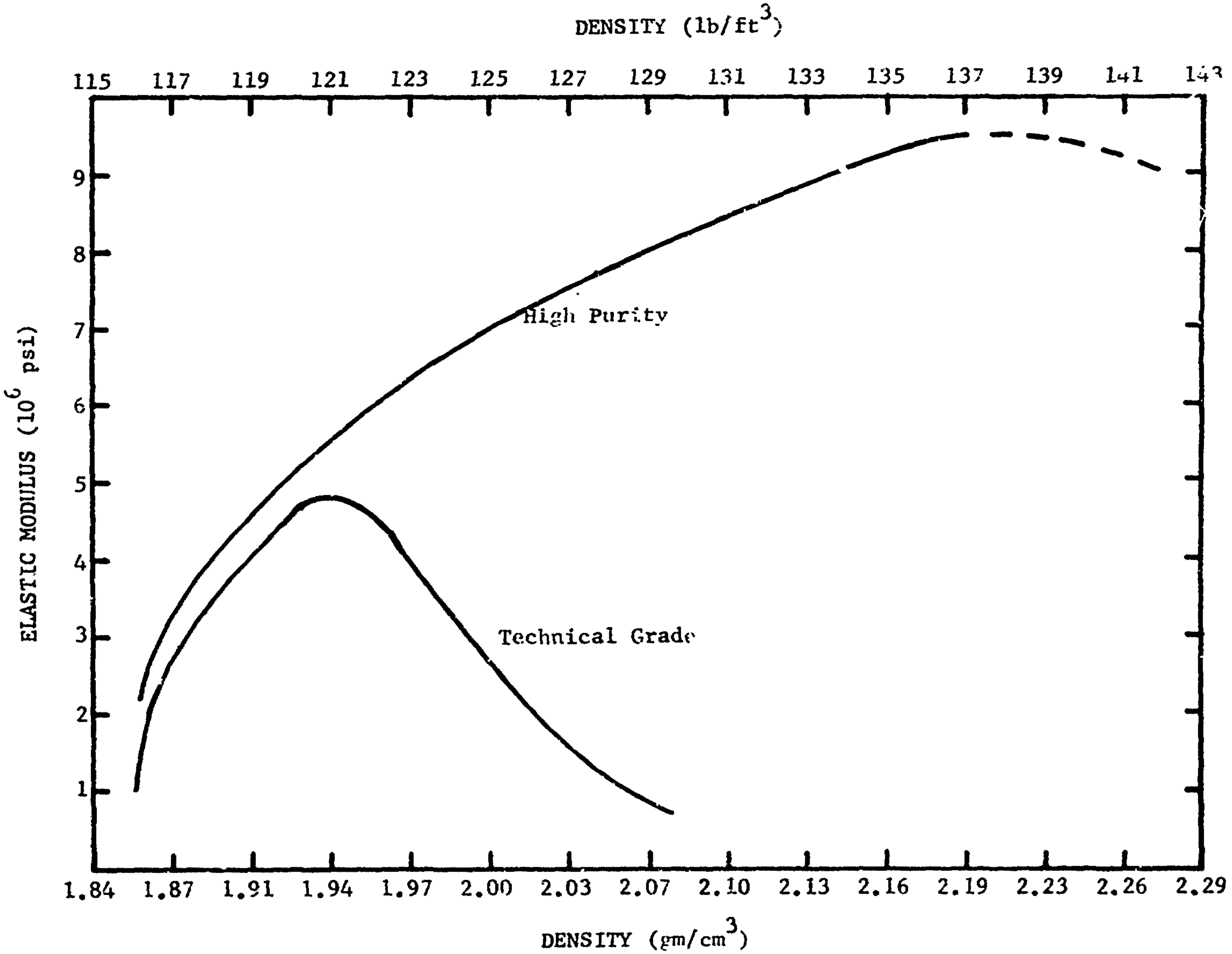

Figure 3. Elastic Modulus versus Density for High-Purity and Technical Grade Slip-Cast Fused Silica. (Reference 5.)

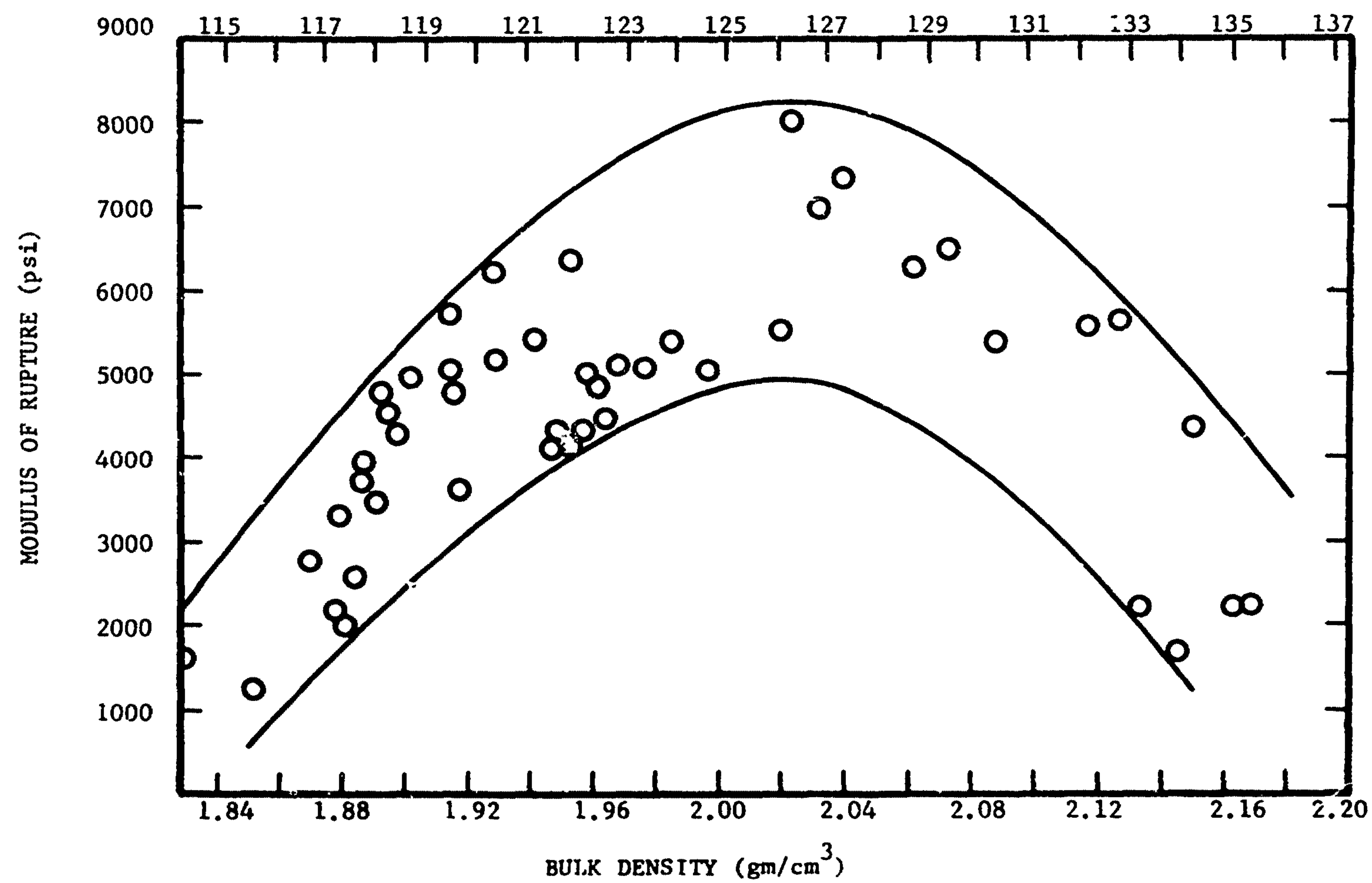

Figure 4. Modulus of Rupture versus Density for High-Purity Slip-Cast Fused Silica. (Reference 5)

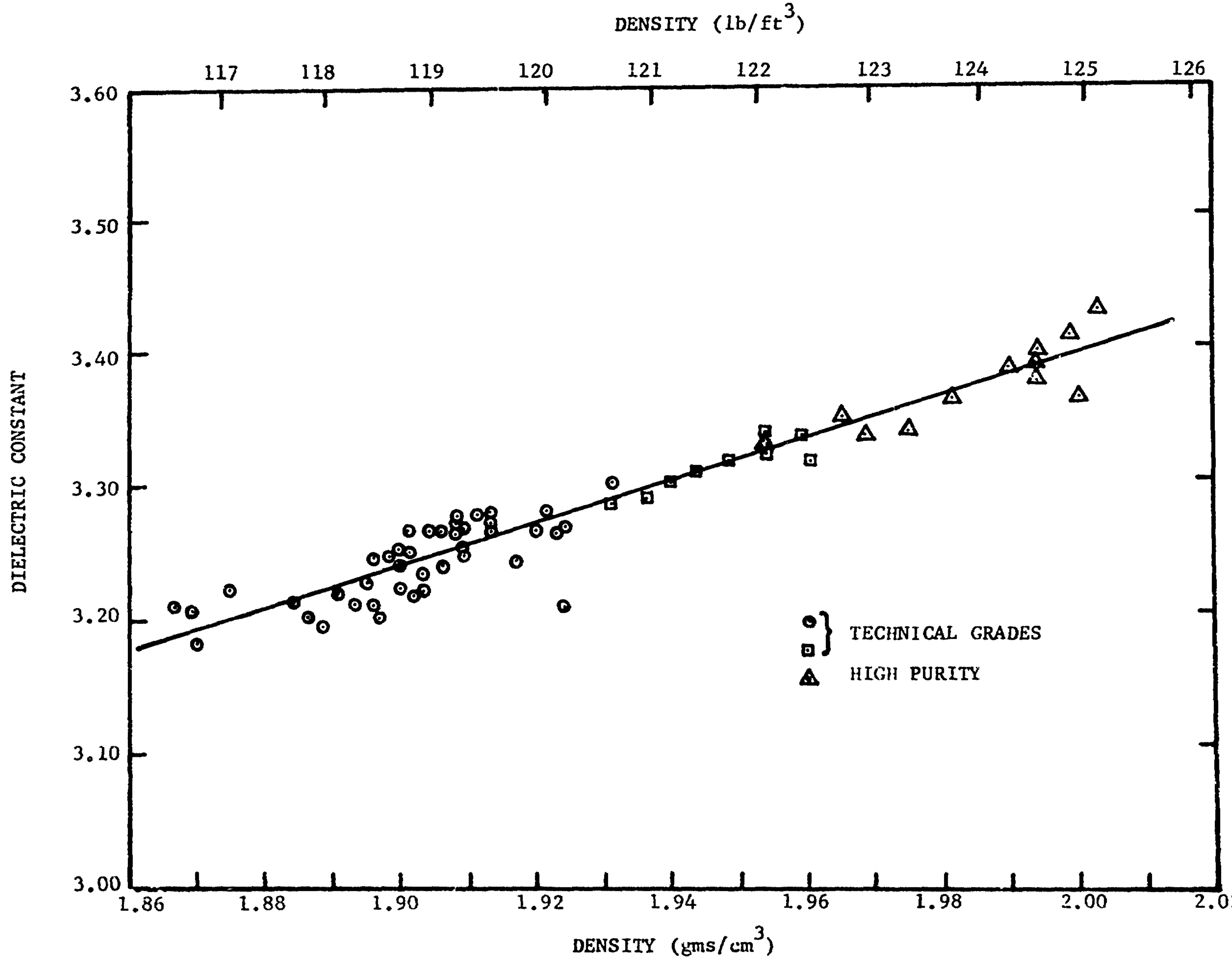

Figure 5. Dielectric Constant of Slip-Cast Fused Silica versus Density Measured at X-Band. (Reference [6].)

per hour must be used to prevent excessive sintering and devitrification of the outside surface of the wall.

H. Machining of Slip-Cast Fused Silica

Although slip-cast fused silica parts can be precision cast to tolerances of the order of ± 0.005-inch 8,9/, some degree of finish machining is often required. Diamond grinding with low (1000-2000 surface feet per minute, sfm) wheel speeds and low material feed rates has proven a satisfactory method of finish machining. Some manufacturers report satisfactory results with dense silicon carbide wheels. Machining should be carried out in such a manner as to prevent contamination of the part. For this reason distilled water is the preferred coolant. In addition, tooling fixtures which come in contact with the silica should be made from non-ferrous materials. Feed rates are limited by the thickness of the part, since comparatively low grinding pressure can cause tensile failure at the back side of the part. Conical shapes with 0.076-inch walls have been successfully machined, but it is felt that this approaches a lower limit on wall thickness. After machining, parts should be dried, and if size permits, leached with dilute (3:1) aqua regia to remove all traces of grinding wheel debris. Drying followed by heating to 1000° F is usually sufficient to remove most organic contaminants such as grease.

NOTE: The above discussion has been directed expressly towards the requirements of the specification attached as Appendix I. For a more comprehensive treatment of slip-cast fused silica radome fabrication techniques and properties the reader is referred to Reference 5.

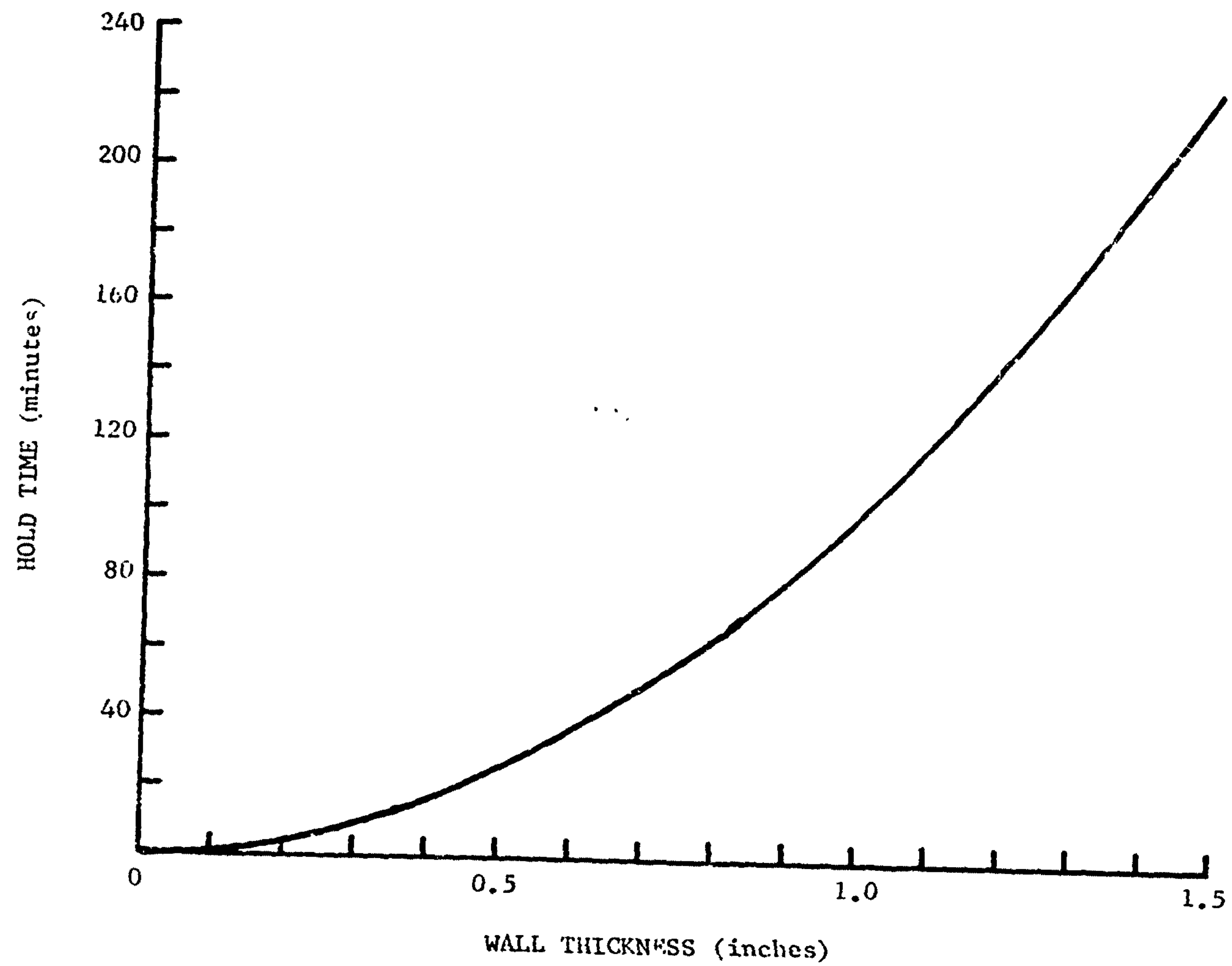

Figure 6. Hold Time at 2000° F Necessary to Raise Backside Temperature to 1990° F, as a Function of Wall Thickness.

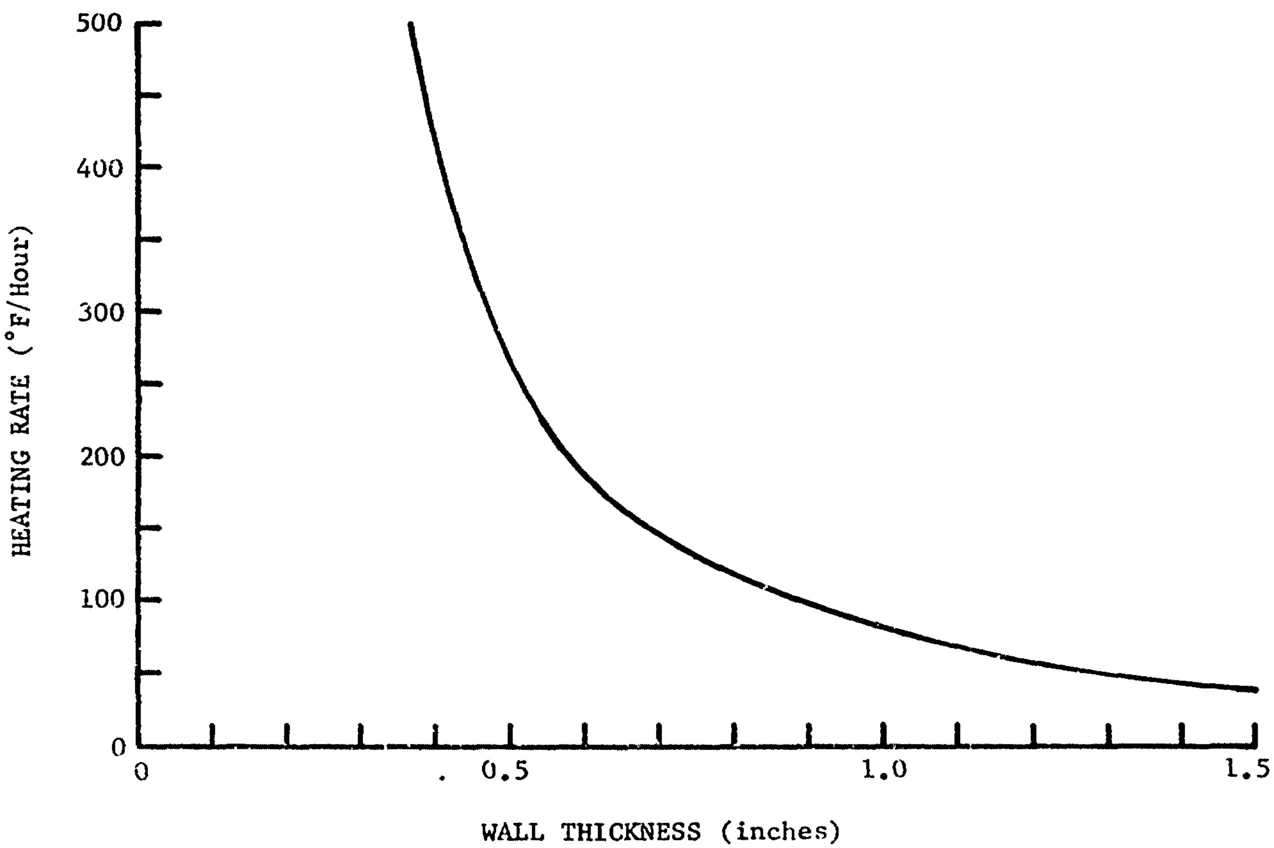

Figure 7. Heating Rate Necessary to Maintain 20° F Gradient Across Radome Wall on Heating from 2000° to 2200° F.

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APPENDIX I

PROCESS SPECIFICATION

Hardware, Slip-Cast Fused Silica

1. SCOPE: This specification covers requirements and guidelines for fabrication of finished hardware from fused silica casting slip.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of initiation for bids or request for proposal, form a part of this specification to the extent specified herein:

SPECIFICATIONS

1. "Fused Silica Slip Requirements for Slip Casting Radomes."
2. "Acceptance Criteria for Slip-Cast Fused Silica Radomes."

3. REQUIREMENTS

3.1 Qualification the material processed in accordance with this specification shall be a product which has been tested and has passed the qualification tests specified herein. Any changes in composition or methods of manufacture of a qualified product shall require requalification as a new product.

3.1.1 Manufacturing Process Procedure. When required by the procuring activity, a titled, numbered, and dated manufacturing inspection document containing the detailed, in sequence operations used in manufacture and control of manufacturing variables, as

specified herein, shall be submitted by the contractor to the Government and its procuring activity for approval before production parts are delivered. After approval, the manufacturing document shall become a part of this specification and copies shall be made available by the contractor for use by authorized personnel from the Government and its procuring activity in the contractor's plant. The manufacturing document shall not be changed without the approval of the Government and its procuring activity.

3.2 Material

3.2.1 Starting Material the starting material for products processed under this specification shall be fused silica casting slip meeting the requirements of "Fused Silica Slip Requirements for Slip Casting Radomes" and "Acceptance Criteria for Slip-Cast Fused Silica Radomes."

3.2.2 Finished Product Finished products processed under this specification shall further satisfy the requirements of the applicable Materials Test Specification.

3.3 Processing

3.3.1 General Processing as specified herein is oriented toward reducing or eliminating contamination of the material with impurities which will contribute to excessive devitrification in further processing or in end use.

3.3.2 Slip Casting

3.3.2.1 Mold Material Plaster of paris shall be an acceptable mold material. Other porous permeable materials such as resin bonded inorganic grain or sintered ceramics shall be acceptable provided products capable of meeting the requirements of "Acceptance Criteria for Slip-Cast Fused Silica Radomes" can be produced.

3.3.2.2 Mold Release Agents Where mold release problems are present, milled graphite slurry or ammonium alginate may be used as mold release agents. Other materials may be used provided that it is demonstrated by comparison of flexural strength of not less than ten test specimens cast with and without mold release agent, that such agent has no significant effect on flexural strength. Flexural specimens shall be in the form of 1/2 x 1 x 5-inch bars broken in quarter point loading, 4-inch span.

3.3.2.3 Casting Slip Storage and Preparation Casting slip will be stored at temperatures above freezing and will be discarded if frozen. The casting slip will be roll agitated for 48 hours prior to use. The slip will be screened to -20 mesh prior to pouring in the mold or slip reservoir.

3.3.2.4 Pressure Assisted Casting Where wall thickness of the cast part is such that it cannot be drain cast within 6 hours, pressure assistance shall be used. Pressure assisted casting shall be acceptable for any cast wall thickness.

3.3.3 Drying Freshly cast parts, particularly large radome shapes shall be dried slowly in the mold until drying shrinkage is complete. At this point the part may be removed and dried in a conventional manner. Cracking of a part in the mold or after removal shall be considered evidence of too rapid drying and insufficient drying in the mold respectively, and will require adjustment of the drying schedule.

3.3.4 Sintering

3.3.4.1 General Sintering will be carried out in such a way as to reach bulk density and strength requirements as required by the applicable acceptance test specification, and in such a way as to minimize thermal gradients within the cast shape.

3.3.4.2 Kiln Requirements Any type of kiln is satisfactory provided temperature over the entire volume occupied by the cast shape can be held within ± 10 degrees Farenheit of the control temperature.

3.3.4.2 Heating Rates To insure minimal thermal gradients in the cast shape prior to final sintering, the casting shall be held at 2000° F for the indicated time according to wall thickness, as follows: up to 0.25-inch - no hold; 0.26-0.50-inch - 30 minutes; 0.51-0.75-inch - 60 minutes; 0.76-1.00-inch - 100 minutes; 1.01-1.25-inch - 160 minutes; 1.26-1.50-inch - 220 minutes. To further insure uniform heat treatment, heating rates from 2000° F to the final sintering temperature shall be no greater than 100° F/hr for parts with wall thickness greater than 0.500-inch, while parts with thinner wall sections may be heated at any rate.

3.3.5 Machining Machining shall be carried out with diamond or silicon carbide abrasives, using distilled or deionized water as a coolant. Jigs and holding fixtures shall be constructed in such a way that only stainless steel or non-ferrous materials shall be in contact with fused silica.

4. QUALITY ASSURANCE PROVISIONS

4.1 Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection and test requirements as specified herein. The Purchaser reserves the right to perform any of the inspections set forth in the specification where such specifications are deemed necessary to assure processing conforms to prescribed requirements.

4.2 Qualification Requirements The qualification of a supplier's process shall consist of meeting all the requirements specified in Section 3 using documentation outlined in Section 4.

4.2.1 Process Qualification Report The supplier shall provide a report showing actual process parameters covering all items required in Section 3.

4.3 Process Acceptance Requirements The acceptance requirements for each lot of material processed shall be all requirements as outlined in Section 3.

4.3.1 Process Acceptance Report For each lot of material processed the supplier shall furnish a report of the process parameters for those items necessary to verify conformance with the acceptance requirements of this specification.

4.3.2 Lot A lot shall consist of those radomes fabricated from one lot of slip.

4.4 Process Documentation

4.4.1 Material The starting material will be noted by vendors lot number.

4.4.2 Casting Each casting shall be identified as to mold, time and pressure of casting, and cast wall thickness.

4.4.3 Drying A log of temperature conditions and drying time will be maintained.

4.4.4 Sintering Schedule A log of time and temperature during sintering will be maintained to insure meeting the requirements of 3.3.4.2.

4.4.4.1 Kiln Characterization A log of time and temperature at furnace centerline elevations corresponding to the top, center and bottom of the casting shall be maintained during the soak period to demonstrate compliance with 3.3.4.1 above. (This requirement for qualification only.)